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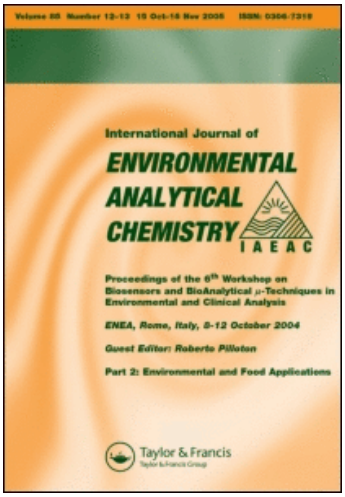

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ALKALOIDS IN ENVIRONMENTAL TOBACCO SMOKE-FILLED ROOMS

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Considering the importance of alkaloids in tobacco smoke, their presence was evaluated in real work environments. Sampling was carried out with fixed pumps and eventual degradation of the alkaloids during collection and storage until analysis was tested. A quantification method with an alternative internal standard (quinaldine) was evaluated and proposed. Offices and leisure rooms with high smoking intensity were chosen in order to detect minor alkaloids. The correlation between the environmental tobacco smoke markers – nicotine, 3-ethenylpyridine and myosmine – and the minor compounds was evaluated and all R^2 were higher than 0.538.

Nicotine levels quantified in the office rooms ranged from 0.02 to 64.67 μg/m^3 and a maximum of 129.33 μg/m^3 was found in one leisure room with very high smoking intensity. Myosmine and nicotyrine were the most abundant minor alkaloids and very high contents were quantified in the leisure room mentioned previously.

Keywords: ETS; Alkaloids; 3-Ethenylpyridine

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INTRODUCTION

Environmental tobacco smoke (ETS) is a highly diluted form of sidestream smoke (the smoke emitted by the burning tobacco between puffs) and of the fraction of exhaled mainstream smoke not retained by the smokers. The most studied compound in ETS has been nicotine but secondary alkaloids are also present in tobacco smoke, so being of interest to examine their appearance in ETS [1,2]. The alkaloids myosmine, nicotyrine and cotinine have been evaluated in particulate and gas phase of ETS in experimental chambers [3–5] and in indoor environments [6]. Since there are few studies to evaluate the different minor alkaloids in real-world situations and also few reports of quantification methods [2,5,7], it is of interest to establish methodologies for their determination and evaluate their presence in realistic conditions.

Due to the chemical complexity and dynamics of ETS in indoor air, selected components have been employed as markers. Nicotine and 3-ethenylpyridine (3-EP) have been

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the most used potential vapor-phase tracers for ETS [8–10]. Nicotine occurs naturally in the leaves of tobacco and when tobacco is burned this alkaloid is transferred to mainstream and sidestream smoke and some is pyrolysed. 3-EP, the predominant isomer of ethenylpyridine formed by pyrolysis of nicotine is detectable in mainstream and sidestream smoke [11,12]. Although in the majority of studies nicotine has been used as marker, of indoor air quality, it may not be the best tracer due to its unpredictable adsorption and decay kinetics originating under or over estimate exposures to ETS [12]. 3-EP has been used as a tracer because its decay characteristics are more similar to those of other ETS constituents, even though it is present in smaller amounts [2,3,5,12,13]. Gas-particulate phase distribution and decay rates of nicotine and 3-EP have been studied in ageing ETS in chambers and experimental rooms [12,14,15].

Several sampling systems for the determination of nicotine and 3-EP in ETS have been tested and compared [16,17]. The most widely used and tested extraction procedure has been the collection with XAD-4 sorbent tubes using modified ethyl acetate [17–20].

Most of the analytical methods for ambient nicotine involve a final determination by gas chromatography and different aspects of nicotine and 3-EP capillary chromatography have been reported [2,17,21].

Quinoline has been used as an internal standard (IS) in the majority of methods for nicotine and 3-EP quantification, but since quinoline has been found in polluted environments, various studies point out the use of high concentrations of this IS (*ca.* 10 μg/mL) to minimize occasional errors [10]. However, this analytical methodology is not adequate, considering that the usual concentrations of nicotine, 3-EP and the other minor alkaloids in ETS extracts occur in much lower levels.

The aim of the present study was to propose an alternative IS not occurring in the real environment, which may consent the evaluation of minor alkaloids in workplace. Different ETS markers, namely, nicotine, 3-EP and myosmine were also tested. In order to permit the detection of these compounds, rooms with high smoke pollution were chosen.

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EXPERIMENTAL

Apparatus

Sampling pumps model 224-50 SKC Ltd. (Blandford Dorset U.K.); Sorbent sampling tubes XAD-4 SKC INC (Pennsylvania, USA); Ultrasonic cleaning bath, Transonic T 660/H, Elma (Germany); Gas chromatograph Hewlett Packard 5890 SII (Wilmington, DE, USA) equipped with a nitrogen–phosphorous detector (NPD), a split/splitless injector, an automatic sampler HP 7673 and an integrator HP 3396 SII; Apolar capillary column CP-Sil8, 5% phenyl, 95% methylsilicone (30 m × 0.32 mm × 1 μm) Chrompack (Middelburg, the Netherlands).

Reagents All reagents were of analytical grade. Nornicotine, anabasine and 4-ethenylpyridine (4-EP) (commercially available isomer of 3-ethenylpyridine) were obtained from Sigma Chemical Co. (St. Louis, USA); myosmine from Carl Roth KG (Karlsruhe, Germany); nicotine, nicotyrine, 2,3′-bipyridyl, quinoline and quinaldine from Fluka Chemie AG (Bucks, Switzerland); cotinine from Aldrich Chem. Co.

(Milwaukee, USA); ethyl acetate, chromatographic quality, from Merck (Darmstadt, Germany) and triethylamine from Riedel de Haen (Seelze, Germany).

Experimental Procedure

An experimental procedure, based on the most widely used, validated and accepted method for nicotine quantification, AOAC method [22] and CORESTA method [23] was adapted for the quantification of minor alkaloids and 3-EP.

Sampling The sampling was established, with the following plan: (1) Indoor air of polluted rooms in Agronomy Faculty was collected; (2) Taking into account the objectives of this work offices and leisure rooms with high smoking intensity (SI) were chosen; (3) Fixed passive pumps (flow $\approx$1 L/min) were localized in the offices at distances of *ca.* 1.5 m from the smokers and *ca.* 1.5 m from the floor; (4) Sampling periods were established between 1 and 2 h.

Compounds Extraction Procedure

The extraction process was carried out in a tobacco smoke free environment. The sorbent sampling tube contents were transferred to autosampler vials for extraction and 1.25 mL of modified ethyl acetate solvent (0.01% triethylamine to prevent any adsorption of nicotine on the glass walls of the vials) containing the IS quinoline (8.0 µg/mL) and the IS quinaldine (0.8 µg/mL) was added to the vials. The vials were then sealed and placed in the ultrasonic bath and agitated for 30 min.

Gas Chromatography Analysis

The gas chromatographic operating conditions were: injector temperature 250°C; NPD detector temperature 300°C; oven program: an initial temperature of 100°C during 4 min, 100–220°C at 4°C min^{-1}; linear velocity of helium 38 cms^{-1}; split 1 : 10; injection volumes of 2–3 µL.

Calibration curves were established using two internal standards simultaneously. Six calibration solutions of standard alkaloids and 4-EP, dissolved in modified ethyl acetate solvent (0.01% triethylamine) with 8.0 µg/mL of IS quinoline and 0.8 µg/mL of IS quinaldine, were prepared. Concentrations from 0.12 to 6.00 µg/mL of nicotine and from 0.05 to 2.50 µg/mL of the 4-EP and the other alkaloids were utilized.

RESULTS AND DISCUSSION

ETS Alkaloids Analysis

Experiments were developed to propose quinaldine as an alternative IS for nicotine and 3-EP quantification in ETS. In order to verify the suitability of quinaldine as IS, experiments were carried out by collecting samples in nonpolluted and polluted smoke environments, in which quinaldine could be detected and none was found. Furthermore, to verify possible interference, from quinaldine impurities, in tobacco alkaloids quantification, blank experiments were developed and it was verified that

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quinaldine, from some suppliers, had impurities that could interfere in tobacco alkaloids analysis. In spite of quinaldine from Fluka having some detectable impurities, these did not interfere in the determination of alkaloids [24]. Figure 1 presents chromatograms of standard alkaloids and an ETS alkaloids extract with the two ISs.

For quantification of nicotine and 3-EP, calibration curves were established with the two ISs. For the other alkaloids, due to their small quantities, the calibrations curves were established only with the IS quinaldine. All the curves presented $R^2 > 0.995$, with the exception of the calibration curve of the 4-EP with the IS quinoline ($R^2 = 0.985$), which can be explained considering the high concentration of the IS quinoline relatively to that of 4-EP. The detection limits (LOD) for the alkaloids were about 0.02 μg/mL with exception of nicotyrine and nornicotine that presented LODs about 0.05 μg/mL.

The accuracy of the method was evaluated through the precision and trueness (Tables I and II). It should be noted that these tests were not conducted with nicotyrine because it was not available. The repeatability of the chromatographic method was evaluated by injecting standard alkaloids and an ETS alkaloids extract five times. The repeatability of the global method, that includes extraction and quantification, was evaluated by conducting five extractions of standard compounds added to sorbent tubes through which free alkaloids air was passed at the sampling conditions chosen for this work. The precision obtained with the IS quinaldine for nicotine and 4-EP was similar or even greater than that obtained with IS quinoline (Table I). In view of this result, subsequent determinations were carried out using the IS quinaldine. The trueness of the method was assessed by extracting the compounds from sorbent tubes spiked with three concentration levels of the different alkaloids and 4-EP (Table II). The lowest recoveries were obtained for the alkaloid anabasine. The accuracy of the improved method is acceptable for the evaluation of 3-EP and the majority of the tobacco alkaloids. For the alkaloid nornicotine heterogeneous results were

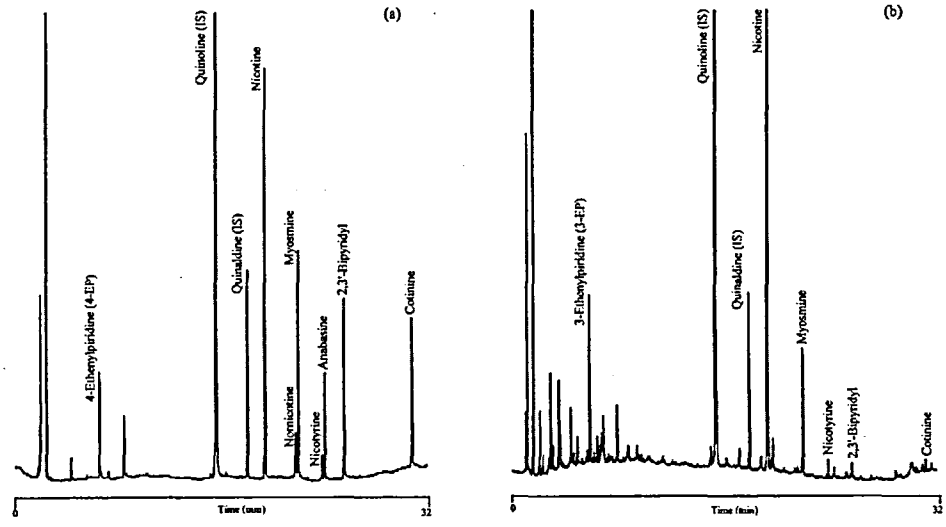

FIGURE 1 Chromatograms of ETS compounds: (a) Standard alkaloids and 4-EP; (b) ETS alkaloids extract.

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TABLE I Precision of ETS alkaloids method*

ETS compounds	*Chromatographic method*					*Global method***	
	Standards			*ETS extract*			
	IS†	*Average* (μg/mL)	*RSD*+	*Average* (μg/mL)	*RSD*+	*Average* (μg/mL)	*RSD*+
4-Ethenylpyridine	(a)	0.39	0.02	0.73	0.01	1.21	0.03
	(b)	0.38	0.04	0.72	0.02	1.26	0.02
Nicotine	(a)	0.84	0.01	2.49	0.01	0.57	0.05
	(b)	0.87	0.02	2.55	0.01	0.66	0.07
Nornicotine	(a)	0.13	0.04	0.37	0.10	–	–
Myosmine	(a)	0.38	0.01	0.18	0.02	0.92	0.02
Anabasine	(a)	0.30	0.00	0.04	0.08	0.40	0.11
2,3′-Bipyridyl	(a)	0.37	0.01	0.06	0.05	0.93	0.02
Cotinine	(a)	0.35	0.02	0.03	0.06	0.85	0.03

* The results are average of 5 replications; **Analysis of standard compounds added to sorbent tubes; †Internal standards: (a) quinaldine; (b) quinoline; +Relative standard deviation.

TABLE II Trueness of ETS alkaloids method*

ETS compounds	*Concentration added* (μg/mL)	*Concentration found* (μg/mL)	*Recovery*	
			%	*RSD***
4-Ethenylpyridine	0.75	0.61	81.3	0.05
	1.19	1.23	103.4	0.03
	2.08	1.95	93.4	0.02
Nicotine	0.35	0.31	88.6	0.02
	0.77	0.74	96.1	0.05
	1.21	1.18	97.5	0.02
Myosmine	0.48	0.44	91.7	0.05
	0.87	0.92	105.7	0.02
	1.38	1.41	102.2	0.02
Anabasine	0.24	0.20	83.3	0.04
	0.50	0.40	80.0	0.11
	0.89	0.70	78.7	0.05
2,3′-Bipyridyl	0.48	0.46	95.8	0.03
	0.85	0.93	109.4	0.02
	1.41	1.42	100.7	0.04
Cotinine	0.42	0.39	92.9	0.13
	0.86	0.85	98.8	0.03
	1.40	1.38	98.6	0.07

*The results are average of 3 replications. Quantification with the IS quinaldine; **Relative standard deviation.

found, probably due to chromatographic and extractive problems and consequently this alkaloid was not considered in the subsequent determinations. It is worth noting that in the more polluted environments, a compound with a retention time similar to that of the alkaloid anatabine was detected in very low quantity.

Considering the possibility of alkaloids degradation during the sampling and storage until analysis, some experiments were developed to check the stability of the different compounds. Although the stability of nicotine collected from ETS has been studied by Muramatsu *et al.*, 1984 [25], preliminary experiments with nicotine and 4-EP were done in order to verify if some occasional degradation of nicotine gave rise to any

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of the studied minor alkaloids. As none of the secondary alkaloids were formed, experiments were developed with all the alkaloids. The procedure was the following: three sets of sampling tubes were used, each tube was spiked with 20 μL of modified ethyl acetate solvent containing the IS and the different compounds with a well known concentration; set (A) was used as reference and alkaloids-free air was passed through the tubes of sets B and C; set (B) was immediately analyzed and set (C) was stored in the freezer (≈4°C) during one week prior to analysis. The results are summarized in Table III and alkaloid's alterations were not observed due to the sampling process or storage of the collected ETS.

Evaluation of ETS in Offices and Leisure Rooms Using a fixed sampling pump, nicotine, 3-EP and minor tobacco alkaloids were evaluated in various office rooms with different smoking intensity (SI). SI is an approach to categorize environments based on the number of cigarettes smoked per unit volume of space and per unit of time. A SI of 0.0244 ± 0.0246 cigt./m^3/h equivalent to 6 cigarettes smoked in a room $40' \times 22' \times 10'$ (226.5 m^3) over one hour, is considered typical [8,26]. In this study 10 offices were selected (Table IV): one nonsmoker office (G) (as a field blank), three offices (J, L, M) with typical SI (0–0.05) and six offices (T, H, P, O, E, Q) with high SI (until 0.30) to ensure a measurable amount of the minor alkaloids. Most of the selected rooms are low height, which contributes to higher SIs. Two leisure rooms were also considered because these are usually environments with high SI. The leisure room R was chosen as an extreme environment condition on account of its small dimensions and the number of cigarettes smoked per hour.

Table V presents the alkaloids and 3-EP concentrations (μg/m^3) in the studied environments. According to Guerin *et al.*, 1992 [8], nicotine in ETS usually ranges from 0 to 10 μg/m^3 and seldom reaches 100 μg/m^3. Levels between 1.1 and 10.0 μg/m^3 and a maximum of 69.7 μg/m^3 were referred in studies of office rooms, reporting 15 or more observations for each particular environment. Nevertheless, some authors [25–27] have found higher values in office rooms, most of them between 9 and 28 μg/m^3. Ogden and Jenkins, 1999 [10] reported recent selected studies which seem to indicate lower

TABLE III Stability of alkaloids on sampler tubes during sampling and storage*

ETS compounds	*Concentration of alkaloids* (μg/mL)						
	Initial	*After sampling*			*After storage*		
			Recovery			*Recovery*	
	A ± SD	*B ± SD*	*%*	*RSD*$^+$	*C ± SD*	*%*	*RSD*$^+$
4-Ethenylpyridine	1.19 ± 0.02	1.23 ± 0.03	103.6	0.03	1.34 ± 0.08	108.7	0.06
Nicotine	0.77 ± 0.06	0.74 ± 0.03	95.5	0.05	0.72 ± 0.06	97.2	0.07
Myosmine	0.87 ± 0.06	0.92 ± 0.02	105.8	0.02	0.94 ± 0.02	102.2	0.02
Anabasine	0.50 ± 0.08	0.40 ± 0.04	80.0	0.11	0.45 ± 0.03	111.4	0.07
2,3'-Bipyridyl	0.85 ± 0.04	0.93 ± 0.02	109.6	0.02	0.93 ± 0.01	99.1	0.01
Cotinine	0.86 ± 0.10	0.85 ± 0.03	98.9	0.03	0.84 ± 0.03	99.2	0.03

*The results are average of 3 replications. Quantification with the IS quinaldine. $^+$Relative standard deviation; A – Quantified after removing from the sorbent tubes; B – Quantified after alkaloids free air was passed through the sorbent tubes; C – Quantified after alkaloids free air was passed through the sorbent tubes and one week of storage.

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TABLE IV Characterization of the smoking environments

Offices identification	*Space dimensions width × length × height*	*Volume* (m^3)	*Ventilation**	*Number of cigarettes smoked/h***	*SI+*
Offices rooms					
G	2.87×3.90 × 2.38	26.64	+	0	0
J	3.57 × 5.00 × 3.48	62.12	++	2	0.03
L	4.15 × 5.05 × 2.30	48.20	++	0.6–1.3	0.01–0.03
M	3.64 × 5.74 × 3.25	67.90	+	1.3–2.5	0.02–0.04
T	3.40 × 3.47 × 3.48	41.06	Air. cond.	0.5–2.7	0.01–0.07
H	3.46 × 3.78 × 2.23	29.17	+++	2.0–2.1	0.07
P	2.74 × 3.63 × 2.04	20.29	+	1.3–4.7	0.07–0.23
O	2.70 × 3.40 × 2.20	20.20	+	3.5–6.0	0.17–0.30
E	1.82 × 4.00 × 2.13	15.51	++	1.3–3.4	0.08–0.22
Q	1.82 × 3.00 × 2.13	11.63	+	0.7–1.5	0.06–0.13
Leisure rooms					
A	5.02 × 6.24 × 2.08	65.16	++	3.3–11.3	0.05–0.17
R	2.86 × 4.83 × 1.69	23.35	+	3.2–22.2	0.14–0.95

*low – +, moderate – ++, high – +++; ** the smoking rate of each occupant during study period; + – SI – Smoking intensity (Number of cigarettes smoked/time unity/volume of space unity).

TABLE V Alkaloids and 3EP concentrations ($\mu g/m^3$) in different ETS natural environments

Offices identification	*Obs. (No.)*	*Number of cigarettes smoked/h*	*Range* ($\mu g/m^3$)					
			Nicotine	*3-EP*	*Myosmine*	*Nicotrine*	*2,3' Bipyridyl*	*Cotinine*
Office rooms								
G	5	0	0.00–0.73	0.00	0.00	0.00	0.00	0.00
J	4	2	0.83–4.65	*	0.00–0.02	*	*	*
L	1	0	0.23	0.00	0.00	0.00	0.00	0.00
	6	0.6–1.3	0.69–6.17	0.45–1.77	0.00–0.50	0.00	0.00	0.00
M	5	1.3–2.5	2.39–10.44	0.74–2.06	0.02–0.35	0.00	0.00	0.00
T	2	0	11.32–16.00	2.10–4.36	0.56–0.82	0.02	0.02	0.32–0.39
	31	0.5–2.7	7.74–64.67	1.24–16.82	0.00–3.45	0.00–3.78	0.00–0.60	0.02–0.50
H	4	2.0–2.1	17.88–21.85	4.09–4.44	1.24–1.51	0.75–1.26	0.02–0.17	0.02–0.29
P	1	0	21.78	4.56	1.21	0.93	0.02	0.29
	3	1.3–4.7	18.23–51.31	4.67–8.18	0.98–2.76	0.02–2.01	0.02–0.48	0.02–0.41
O	4	0	0.00–6.09	*	0.00	*	*	*
	10	3.5–6.0	32.21–119.34	*	2.09–6.18	*	*	*
E	4	0	0.02–0.87	0.00–0.02	0.00–0.06	0.00	0.00	0.00
	7	1.3–3.4	0.02–27.42	0.00–6.62	0.00–1.70	0.00	0.00	0.00
Q	2	0	0.02–1.27	0.00–0.59	0.00–0.09	0.00	0.00	0.00
	5	0.7–1.5	5.99–26.34	1.83–6.58	0.37–1.06	0.00–0.92	0.00	0.00
Leisure rooms								
A	8	3.3–11.3	4.58–82.84	1.36–13.62	0.37–4.71	0.00–3.93	0.00–0.69	0.00–0.59
R	2	0	8.11–9.95	2.74–3.30	0.26–0.29	0.00	0.00	0.00
	11	3.2–22.2	31.00–129.33	8.06–27.58	1.12–6.78	0.00–5.67	0.00–1.23	0.00–0.02

*Compounds not quantified; For trace compounds a 0.02 value was assumed.

nicotine levels. As shown in Table V, in the office rooms with typical SI (J, L, M), data appraised for nicotine were similar to values usually referred to in different studies [8,10]. However, in the majority of the cases, the nicotine levels were higher in office rooms (0.02 – 64.67 $\mu g/m^3$) and in leisure rooms (4.58 – 129.33 $\mu g/m^3$). The later results may be explained by the low height and deficient ventilation of the selected rooms

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TABLE VI ETS alkaloids levels (μg/m^3) in occupied rooms and their correlation with the ETS markers*

ETS compounds	*Obs. (No.)*	*Mean ± SD*	*Median*	*Range*	*Nicotine/comp.*+		*3-E/comp.*+		*Myosmine/comp.*+	
					R^2	*Ratio*	R^2	*Ratio*	R^2	*Ratio*
Offices rooms										
Nicotine	55	23.18 ± 17.14	20.28	0.02–64.67	1.000	1.000	0.931	0.206	0.933	0.046
3-EP	55	5.13 ± 3.66	4.44	0.00–16.82	0.931	4.517	1.000	1.000	0.859	0.206
Myosmine	55	1.16 ± 0.81	1.15	0.00–2.86	0.933	20.323	0.859	4.163	1.000	1.000
Nicotyrine	55	0.79 ± 1.00	0.02	0.00–3.78	0.717	14.548	0.665	2.991	0.613	0.640
2,3′-Bipyridyl	55	0.14 ± 0.19	0.02	0.00–0.60	0.729	78.038	0.593	15.027	0.644	3.485
Cotinine	55	0.16 ± 0.18	0.02	0.00–0.50	0.622	75.643	0.578	15.580	0.569	3.439
Leisure rooms										
Nicotine	19	61.83 ± 39.40	72.33	4.58–129.33	1.000	1.000	0.944	0.202	0.792	0.043
3-EP	19	12.72 ± 8.19	12.15	1.36–27.58	0.944	4.675	1.000	1.000	0.818	0.209
Myosmine	19	2.79 ± 1.89	2.32	0.37–6.78	0.792	18.567	0.818	3.920	1.000	1.000
Nicotyrine	19	1.64 ± 2.16	0.02	0.00–5.67	0.538	13.405	0.579	2.890	0.679	0.722
2,3′-Bipyridyl	19	0.31 ± 0.45	0.02	0.00–1.23	0.599	67.256	0.685	14.948	0.860	3.861
Cotinine	19	0.07 ± 0.17	0.00	0.00–0.59	n.c	–	n.c	–	n.c	–

*The observations done in offices O and J and some of the office T were not included. For trace compounds a 0.02 value was assumed; +Compounds quantified in ETS; n.c. – Not correlated.

(H, P, O, E, A, R). In addition their occupants were heavy smokers. In spite of the greater dimensions of the office room T, very high concentrations for the studied compounds were also found. Peculiar conditions in this office (damaged air-conditioning, and the excess of furniture and the position of the sampling pump near a bookcase) can explain these high levels. High alkaloids and 3-EP levels were also found in two samples collected in the absence of the smoker (SI=0), this fact indicating that it is not correct to establish a direct relation between the SI and the ETS compounds. A similar situation was observed in offices P and O (Table V). The walls and furniture surfaces may eventually be coated with nicotine, which may be re-emitted to the air. It is to be mentioned that these results are consistent with other studies [15,28]. Assuming that nicotine adheres more tightly to the wall surfaces than to stainless steel chambers, this assumption can explain the discrepancies between nicotine levels in chambers and in real environments, where smoking occurred regularly [29,30].

In Table VI are presented the ETS alkaloid levels and their correlation with the ETS markers – nicotine, 3-EP and myosmine. The three markers coefficient determinations *vs* the compounds studied were greater than 0.536 and in principle they can be used to predict the presence of minor tobacco alkaloids. The ratio of nicotine to 3-EP was *ca.* 4.5 and to myosmine *ca.* 20. Cotinine was not detected in environment R and consequently this alkaloid was not correlated with the ETS markers in the leisure rooms (Tables V and VI). A straightforward explanation for this fact cannot be easily found. In the observed environments, myosmine and nicotyrine were the most abundant minor alkaloids, the highest values having been quantified in the leisure room *R* (Tables V and VI).

Considering the correlations of 3-EP and nicotine with the minor alkaloids, both are good markers. Taking into account that the 3-EP concentration is more in accordance with the minor alkaloids concentration in ETS, we consider the use of 3-EP as a more convenient marker.

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